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PRELIMINARY INVESTIGATION OF LOW TEMPERATURE PHASE TRANSFORMATIONS IN AISI 301

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INTRODUCTION:

A preliminary investigation has been conducted on the martensitic transformation in AISI 301 at cryogenic temperatures. This program was undertaken in view of failures of Centaur bulkheads in weld areas.

It is well known that 300 series austenitic stainless steels undergo a strain induced martensitic transformation from face-centered cubic austenite (γ) to body-centered cubic martensite (α'). A less well-known fact is that martensitic transformation can occur in certain 300 series stainless steels[1] spontaneously upon cooling to the vicinity of -320°F. In addition, a close-packed hexagonal transformation product (ϵ) transitional between γ and α' has been recently identified.[2, 3, 4]

The experimental techniques employed in this investigation and certain of the assumptions made were based on the results of an investigation* conducted at the Cryogenic Engineering Laboratory, National Bureau of Standards to evaluate the effect of the martensitic transformation on the properties of austenitic stainless steels[1, 3, 5, 6, 7].

OBJECTIVE:

The objective of this program was to determine the possible influence of martensitic transformations in AISI 301 on the low temperature service failures of Centaur bulkheads.

SUMMARY AND CONCLUSIONS:

1. It is concluded that a spontaneous martensitic transformation occurs in annealed AISI 301. The magnitude of the transformation varied from zero to in excess of 50 per cent depending on the chemistry of the particular heat tested.

*Sponsored by the Advanced Research Projects Agency of the Department of Defense.

2. A close-packed hexagonal phase (ϵ) transformation product which appeared to be transitional in nature was found in AISI 301 annealed. This phase was found to occur both spontaneously and as a function of strain.

3. The spontaneous transformation that occurs in annealed AISI 301 base material also was found in spot welds of cold-rolled AISI 301.

Based on previous work and the results obtained in this program, the opinion was reached that AISI 301 is unsuitable for use in the spot weld configuration under the present standards and selection criteria.

RECOMMENDATIONS:

1. It is recommended that AISI 301 not be used in the spot weld configuration until stabilization against spontaneous martensitic transformations to an acceptable degree be accomplished.

2. Stabilization can possibly be accomplished by controlling the chemistry within nominal compositional limits. Increased C, N_2 and Ni content will promote stabilization, although with the increased C and N_2 content you increase the deleterious effect on the fatigue properties[9, 10].

3. Small pre-strains at ambient temperatures or above promote transformation while large pre-strains promote stabilization. In addition, during strain-induced transformation at room temperature, the ϵ phase can be expected to be largely suppressed. Therefore, a pre-strain of the weld may attain a desired degree of stabilization.

SPECIMENS:

All specimens were cut from cold-rolled AISI 301

Stainless Steel Stock

Small Strain Test Specimens

Standard 9 inch tensile coupons were cut from cold-rolled AISI 301 Sheet Stock, Heat Number 62879.

Compositional Fluctuations on Spontaneous Transformation Specimens

Specimens were cut from seven different heats (See Table I) in the form of 1 x 3 inch blanks.

Fusion Weld Specimens

1 x 3 inch blanks from Heat Number 57644 were fusion welded together to form the final specimens.

PROCEDURE:

Specimens of cold-rolled AISI 301 were annealed and subjected to temperature cycling tests between room temperature and -320°F. Small amounts of tensile strain were introduced at -320°F. in some of the specimens. Metallographic, magnetic and X-ray diffraction examinations were conducted. The magnetic study employed a magna-gage that had been pre-calibrated to measure percent α'. X-ray traces were obtained with a horizontal diffractometer, a counter and a scalar circuit in conjunction with a recorder.

Small Strain Procedure

The specimens were annealed at 1850°F. and air cooled. Three specimens were strained at -320°F. 2.5%, 5%, and 9% in a two-inch gage length respectively. All of the specimens were descaled by Electro-polishing. The specimens were then scanned from 60 to 90 degrees with the X-ray diffractometer in two separate areas.

Compositional Fluctuations Procedure

One specimen from each heat was annealed at 2000°F. and air cooled. The specimens were then electropolished to remove heat-treating scale and subjected to temperature cycling in LN_2. The spontaneous transformation to α' was measured magnetically.

Fusion Weld Procedure

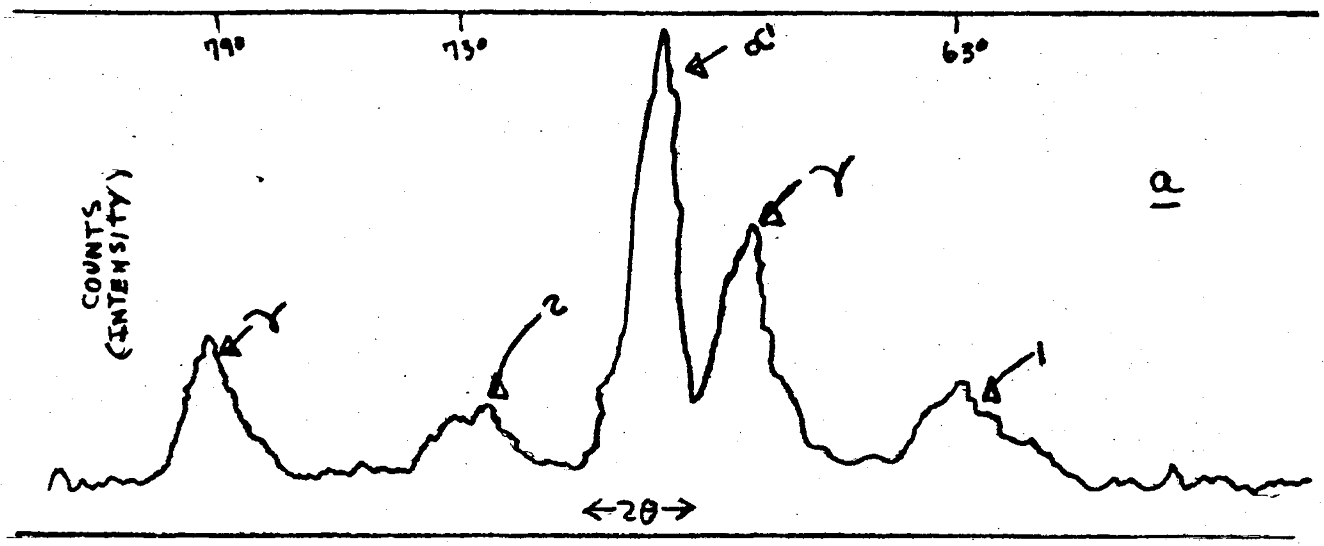
Specimens were cut from two weld cross sections, mounted, mechanically polished and lightly etched.

A metallographic study was then made. It should be noted that a final electrolytic polish is needed for maximum accuracy of results.

RESULTS:

Small Strain

X-ray diffractometer patterns revealed the presence of ϵ as well as α' phase. Small peaks were verified by counting techniques.



Peaks labeled 1 and 2 above represent reflections from the close-packed hexagonal ϵ phase[4]. The specimen strained 2.5% exhibited the largest peaks for the ϵ phase. The annealed specimen evidenced no detectable ϵ reflections and the reflections for the 9% strained sample were detectable only through scalar counting. The annealed specimen was subsequently cycled in LN_2 and the X-ray trace revealed both ϵ and α' phase reflections.

Compositional Fluctuations

The spontaneous transformations to α' were measured with a magnagage and found to be as follows:

MAGNA GAGE READING

Before Quenching Specimen	Avg. % α'	After One Cycle 5 Min. at -320°F. Specimen	% α'	After Four Cycles 5 Min. at -320°F. Speci.	Avg.% α'
A	0	A	1	A	4
B	0	B	48	B	48
C	0	C	24	C	43
D	0	D	21	D	38
E	0	E	26	E	30
F	0	F	0	F	0
G	0	G	45	G	48

The increase in transformation with cycling is consistent with previous work on 300 Series Stainless Steels[1, 6]. In order to recheck the results, another group of specimens were annealed, descaled, quenched and qualitatively checked for magnetism.

Specimens A and F evidenced no detectable magnetism and the rest were magnetic to varying degrees.

X-Ray diffraction traces were made for specimens B, D, E and F and are illustrated in Figures 1 through 4 respectively. The various reflections are identified on the traces.

Fusion Weld:

Figures 5 through 7 are micro-photographs of one of the weld specimens. Figure 5a represents an area including both the weld bead and the heat affected zone as welded. The crack (upper left) is the junction of the two joined sheets. The striations in some grains (note area A) and surface upheavals represent strain-induced transformation caused by the final stages of mechanical polishing. Unless dislocations are etched, after the fact etching will not delineate either slip lines or surface deformation due to α'. Figure 5b represents the same area after quenching and holding for 10 minutes at LN_2. Propagation of existing plates and new formation are well illustrated near the twinned γ grain. Area B illustrates a new state in the heat affected zone and area C a new state in the weld bead.

Martensitic transformations are known to be heterogeneous. This was evident in examining both specimens. Figure 6 represents a heavily transformed area in the heat affected zone of a specimen after quenching. Notice the apparent crack. In examining the specimen prior to quenching, nothing of this nature was detected. The discontinuity was examined at different magnifications under varied lighting and was judged to be a crack rather than a ferrite stringer. Figure 7 is a view of the discontinuity in relation to the weld bead at left and the base material extreme right.

DISCUSSION:

The time available for this program and report was extremely limited. Therefore, the program was conducted to obtain the maximum information in a minimum time. Undoubtedly, the use of more time consuming techniques and more experiments would add considerable information to the results presented in this report.

Spontaneous formation of α' is accompanied by Approx. 4 percent volume increase per unit volume transformed. Therefore, spontaneous formation of α' can be assumed to result in localized residual stresses. The magnitude of these stresses would be dependent on the amount of γ transformed and the ability of the parent material to distribute stress concentrations. As percent γ decreases, stress relief around stress concentrators becomes increasingly difficult. Residual stresses coupled with the fact that brittle ϵ phase forms spontaneously in annealed AISI 301 leads to the supposition that a high probability exists for the initiation of cracks in certain weld zones at low nominal service stresses. Many service failures that have occurred in the past in brittle materials at nominal service stresses below the yield are attributed to residual stresses[8].

It is logical to expect that the results presented here would not be detectable in standard mechanical property tests on cold-rolled AISI 301. Even cracks formed in weld areas would not be expected to propagate catastrophically as in the case of wholly brittle structures. As a matter of interest, laboratory tests have been notoriously unsuccessful in duplicating "in service" failures.

In a completely stabilized AISI 301 weld residual stresses due to α' formation would be eliminated and in addition, no ϵ would form until service stresses produced localized plastic strains.

Along the lines of the preceding discussion, it is believed that AISI 301 is unsuitable for structural purposes in the spot welded condition without control of the spontaneous martensitic transformations. It is noteworthy that the strain-induced transformation characteristics of AISI 301 are analogous to 18-8 austensitic stainless steels and they should exhibit similar service behavior with AISI 301 in the stabilized condition.

TABLE I

CHEMISTRY OF SMALL STRAIN AND FUSION WELD

Type Specimen	Heat No.	C	N_2	Cr	Ni	Mn
Small Strain	62879	0.07	0.03	17.3	7.4	1.27
Fusion Weld	57644	0.08	0.035	17.4	7.25	1.00

TABLE II

CHEMISTRY OF SEVEN HEATS USED IN THE COMPOSITIONAL FLUCTUATION ON SPONTANEOUS TRANSFORMATION

Specimens	Heat No.	Chemistry				
		C	N_2	Cr	Ni	Mn
A	48646	0.05	0.04	17.07	7.25	1.00
B	49287	0.07	0.03	17.2	6.80	0.70
C	49001					
D	49324	0.07	0.04	17.3	6.8	0.71
E	49095	0.09	0.03	17.7	6.7	0.72
F	47156					
G	49061					

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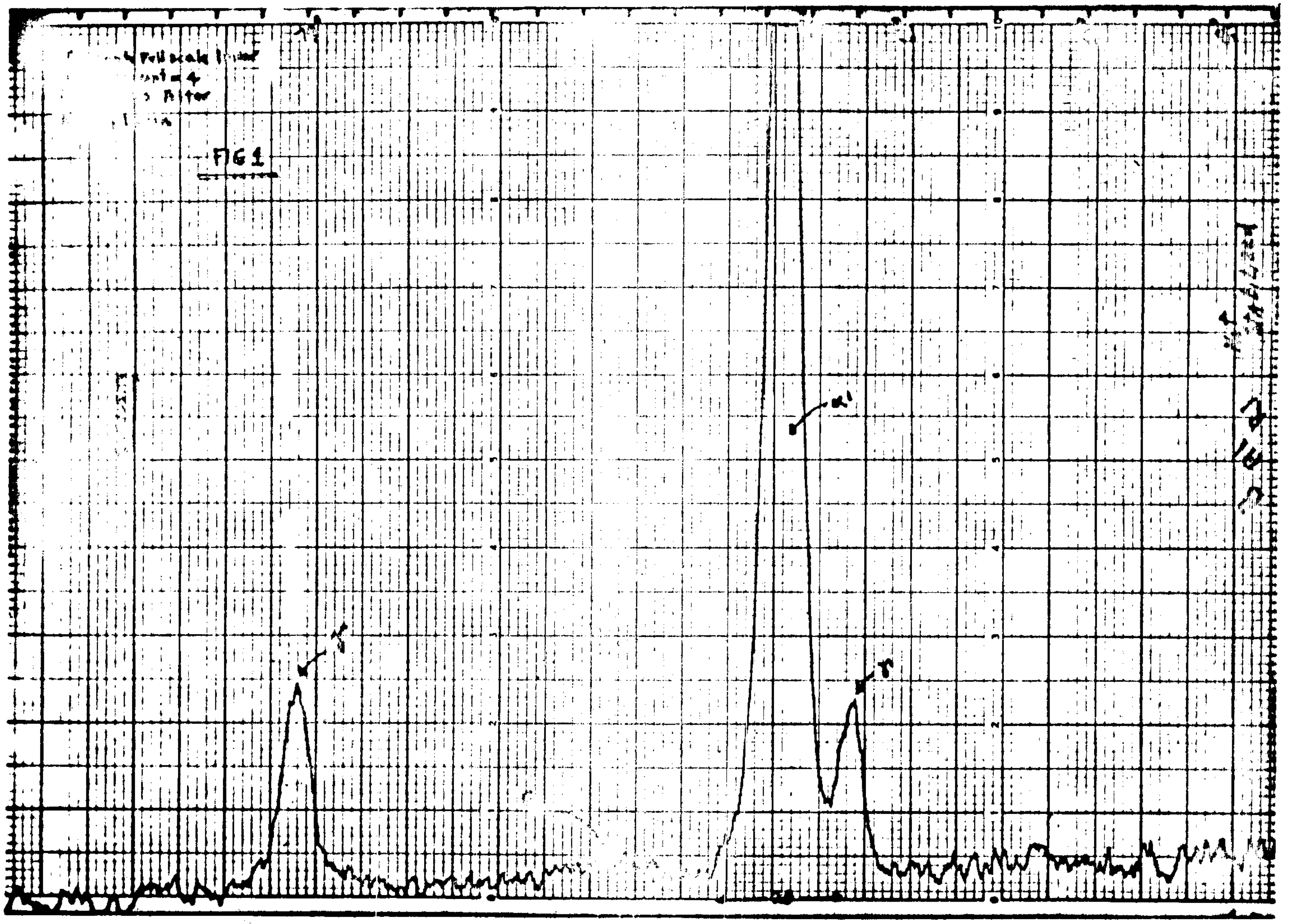

Figure 1

M 6749, Annealed at 2000°F. Air Cooled Cycled in LN_2
Specimen B, Area 1, Run 1

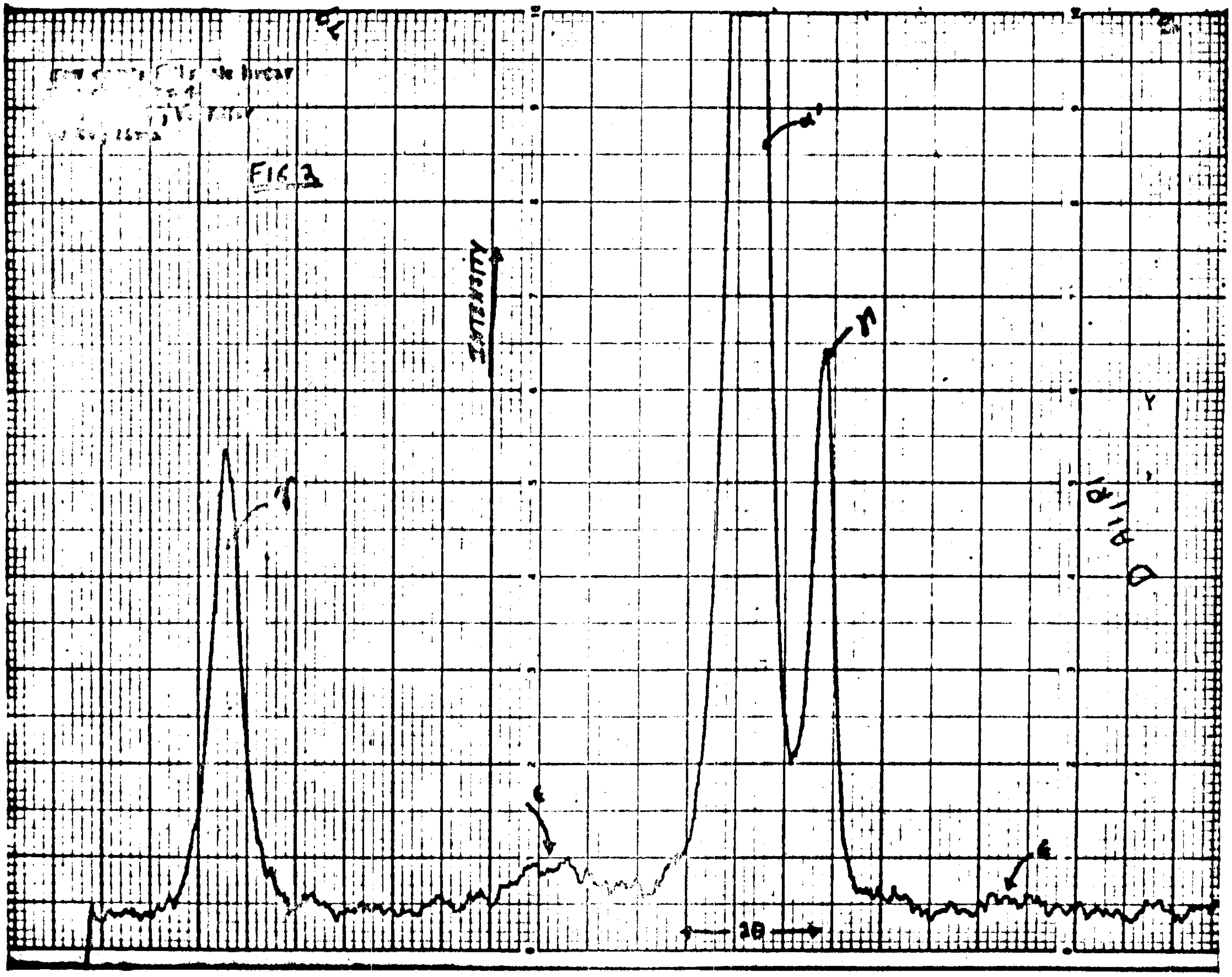

Figure [illegible]

M 6748, Annealed at 2000°F. Air Cooled Cycled in LN_2

Specimen D, Area 1, Run 1

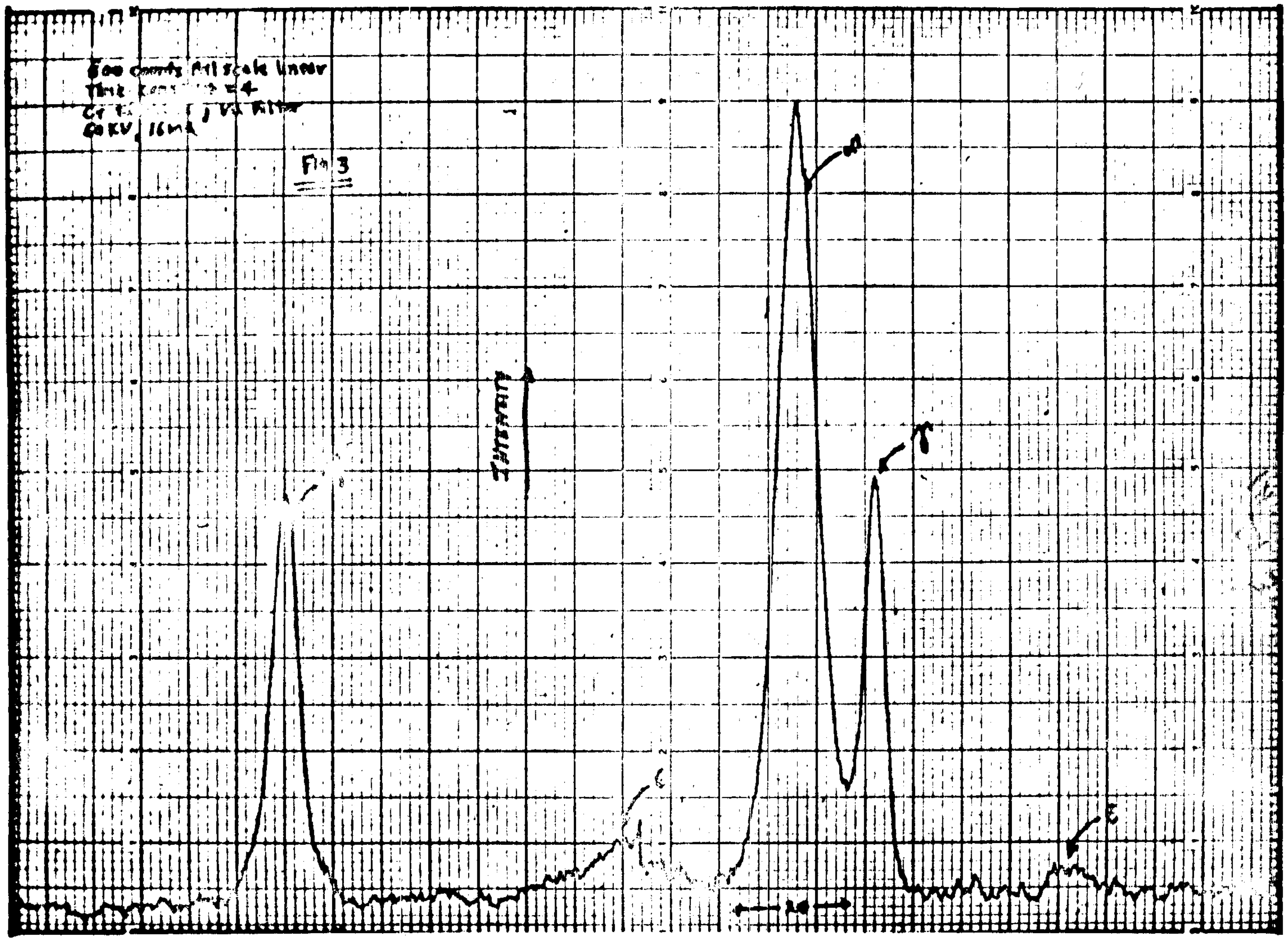

Figure 3

M 6747, Annealed at 2000°F, Air Cooled Cycled in LN_2

Specimen B, Area B, Run 1

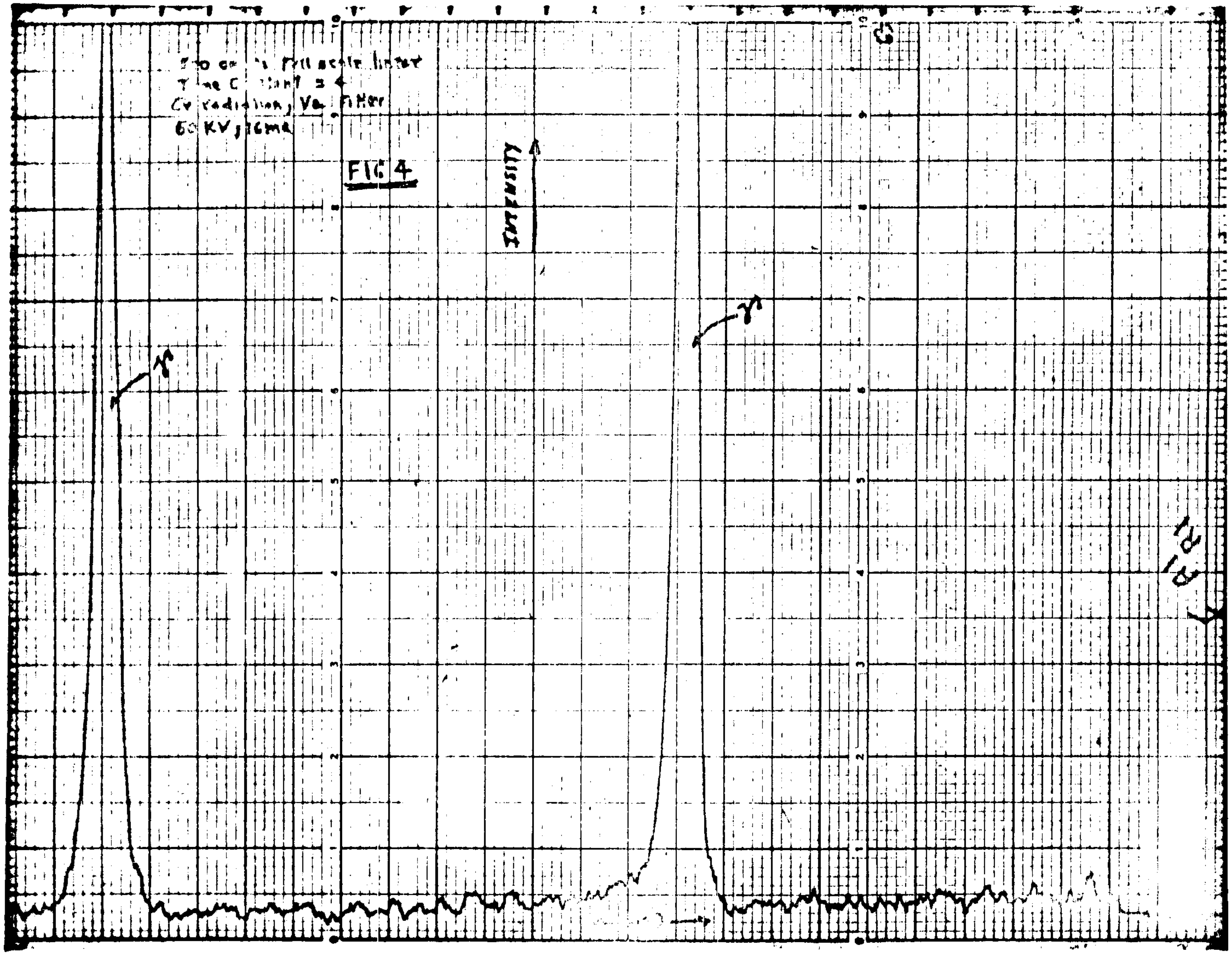

Figure 4

M 6746, Annealed at 2000°F. Air Cooled Cycled in LN_2

Specimen F, Area 1, Run 1

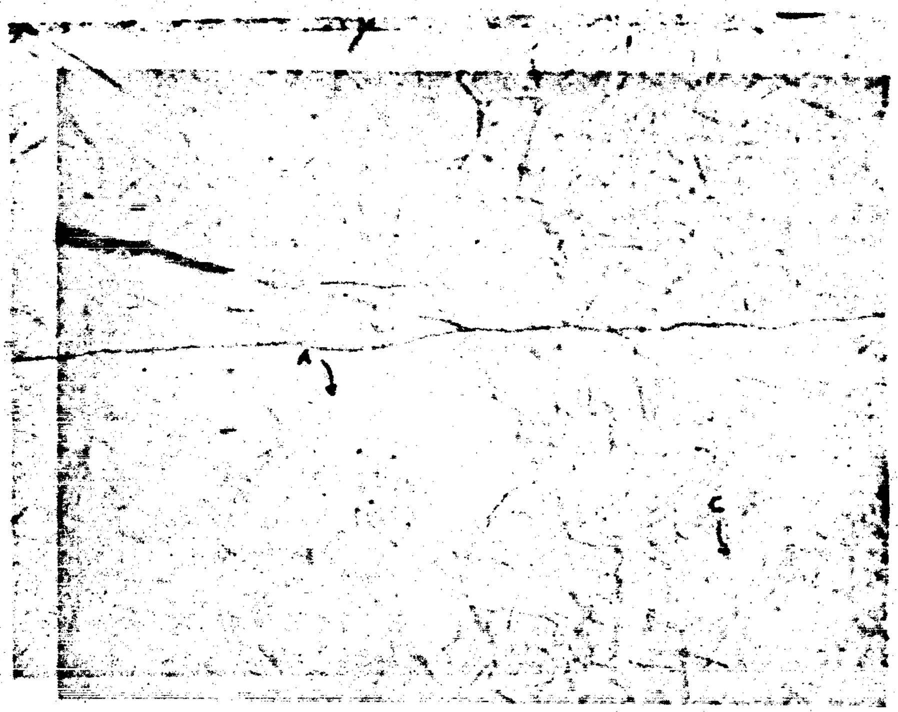

Figure 5a

AISI 301 Spot Weld, 1000X, Electro Etched 10% Oxalic

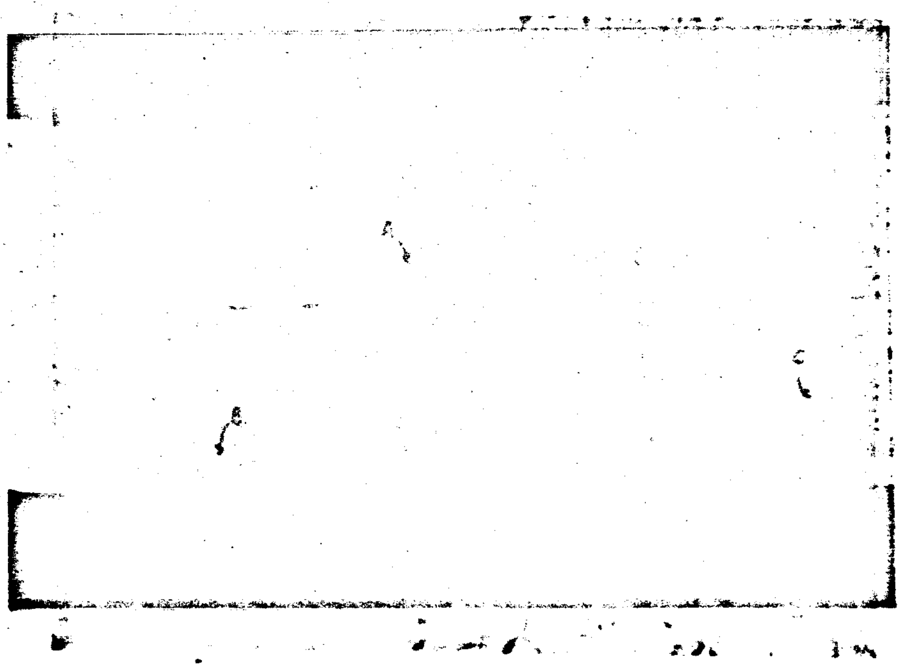

Figure 5b

AISI 301 Spot Weld After Cycling to LN_2, 1000X

Figure 6

Apparent Crack in AISI 301 Spot Weld After Cycling to LN_2, 500X

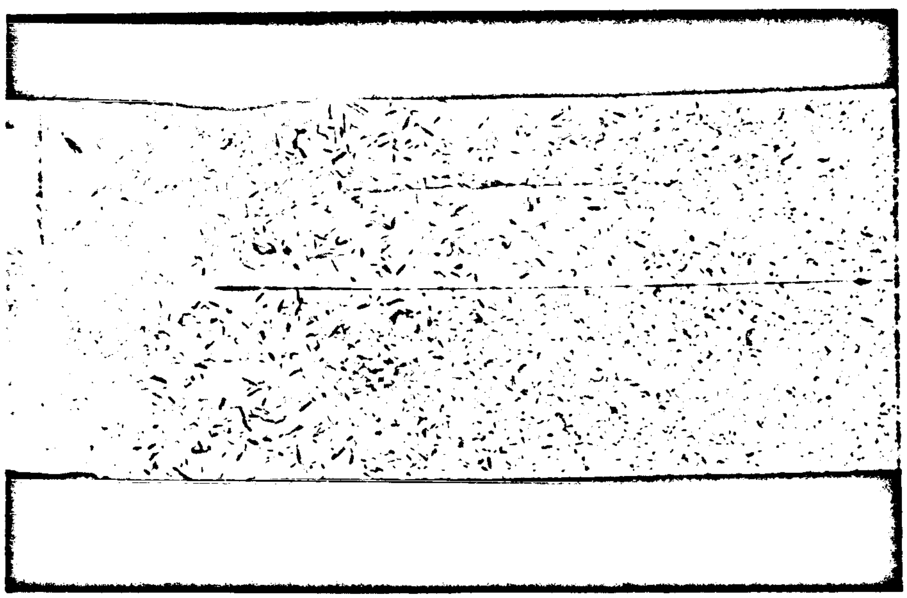

Figure 7

View of Spot Weld Crack in Relation to Weld Bead, 100X